

Appn. Number 10/653,678 Michael John Keogh Nguyen/2831 Amnt.E

Requested Correction on Wording of Claim 47

Claim 47 was corrected as required.

Rejection of Claims 35-39 and 42-51 Under 35 U.S.C. 103(a)

Claims 35-39 and 42-51 were rejected under 35 U.S.C. 103(a) as being unpatentable over Kim et al. (2002/0096356) in view of Weil et al. (5,578,666).

Applicant asserts that at least three major differences distinguish his application from the teachings of Kim et al. and renders his invention outside the U.S.C. 103(a) objection.

First- Claims 35-39 cover technology targeted to satisfy jacket/sheath requirements in cable construction. Jacket requirements differ substantially from the Kim et al. insulation requirements.

Second- Kim et al. is for an insulation component. As such, the properties required reflect those for this application area. Mechanical properties, such as cut through required Kim et al. to cross-link the inner layer of his system. Cross-linking, discussed separately below, is the greatest distinguishing feature between the applications.

Third- Mechanical properties led Kim et al. to the choice of a Nylon 11 based resin system. Kim et al. is not aware that his inner FR layer, based on metal hydrate FR technology, would benefit greatly from an outer thermal barrier layer as a replacement for the nylon. Mechanical property retention trumps any consideration to increase FR in the outer layer.

Thermoplastic vs. Thermoset in Wire and Cable (Differences 1st and 2nd)

Every physical, mechanical, chemical, electrical and flame property of a resin is substantially changed in going from the thermoplastic to the thermoset state. Kim et al. chooses to add a cross-link process step and the added cost to meet requirements.

In Amendment D (12/05/2005) applicant detailed why Kim et al. was required to go to a thermoset resin system. Briefly the mechanical and electrical requirements for his insulation composition dictate the need to cross-link. Applicant's jacket/sheath application does not require this added modification and process step.

Applicant has amended claims 35 and 36 to include the designation thermoplastic to highlight this significant distinguishing feature of his invention.

Outer Layer Composition (Third Difference Discussed)

Examiner states Kim et al. does not disclose his outer layer as being intumescent. Further, examiner states that one skilled in the art would use Weil et al. (5,578,666) for the outer layer to enhance the flame retardance of the insulation system.

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The choice of an intumescent thermal barrier protective system would only be made by a researcher that had grasped the idea that the FR efficiency of the inner layer hydrate FR system was dependent on thermal insulation. Neither Kim et al. nor any other skilled in the art had made that connection prior to applicant. Applicant has presented, in the subject application [0054] and in Amendment B, pg.5, Review of Key Features of Invention (Nov. 15, 2004), the reasoning that led to his novel construction. For many years those skilled in the art were fully aware of both the need for flame retardance and the specific FR technologies but they never conceptualized applicant's unique combination as a solution. In support of this statement, Professor Weil et al. (5,578,66), an expert in the field, views his development as a composition for increased flame retardance but does not address the thermal insulating possibilities. Others skilled in the art have always attempted to improve flame resistance in hydrate systems by increased loading of metal hydrate. This approach has serious limitations and is abandoned at the point where the loss of properties outweighs the gain in FR.

Applicant believes his invention is novel and no prior art shows awareness of the factors behind his development. Choosing an intumescent layer would be by chance and certainly not obvious. In summary "only applicant has connected the dots."

Examiner in the "Claims Rejection" section and again in the "Response to Arguments" section cites Kim et al. as disclosing that his inner layer could be a "polypropylene copolymer". Specifically, Kim et al. mentions ethylene-propylene thermoplastic elastomer as resins for cross-linking. Polymer chemists view EPR elastomers to be ethylene and not a propylene copolymers. The reasoning here is based on ethylene being the comonomer in highest concentration. Propylene is added as a comonomer in order to reduce the crystallinity of the polymer system. In this way a material that has elastomeric (rubber) and not plastic properties is produced.

Applicant does not consider the Kim et al. citation of EPR as a resin precursor for cross-linking to be significant. Nevertheless, applicant for added clarification amended claims 36,38,43,44,45,47 and 49 to describe more precisely the propylene copolymer used in the invention.

Re claim 42 and the suggested use of the Kim et al. conductor in the claim. Applicant does not see a connection. Kim et al. always employs a thermoset resin system. Substituting applicant's homopolymer or copolymer of propylene to afford a radiation cross-linkable system is not a viable approach. Applicant's propylene-based polymers undergo degradation over cross-linking when exposed to the Kim et al. radiation cross-linking process. Polypropylenes are one of a number of polymers never recommended for direct cross-linking. (Attached is a literature reference showing the

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problems encountered with PP cross-linking.)

Re comments on claim 49.

Applicant has argued at length against Kim et al. as suitable prior art. Re the claim 49 itself, applicant has made the point that researchers skilled in the art, although aware of the hydrolysis and subsequent corrosion problems encountered with the use phosphorous-based intumescent, never conceptualized applicant's solution. For example, Weil et al. mentions that phosphorous systems present hydrolysis and corrosion problems (see Weil et al. 5,578,666, col.1 line 65- col. 2 line 5) He does not however attempt to suggest any solution.

Any quantity of acid produced and not neutralized will lead to conductor corrosion. This is never acceptable in a wire and cable insulation compound. Simple wire discoloration alone will eliminate a system from consideration. Applicant's placing the neutralization agent in the insulation layer between the acid source and the corrosion target is novel and has escaped those skilled in the art. Examiner's suggesting that it would be obvious to adjust the level of FR additive to accomplish the neutralization assumes facts not in evidence. First the construction would have to be applicant's to exhibit the problem. The researcher would have to realize that a corrosion problem from acid released in the outer layer was causing the corrosion, Finally the connection that the magnesium salt would serve to neutralize the acid would have to be made. In hindsight this may be obvious but the fact still remains that before applicant no one had seen the solution. It is not a simple matter of determining an optimum or workable range that is important.

Additional Comments on Response to Arguments Section of OA.

Examiner states that applicant argued "there is nothing in Kim et al. to suggest that his thermoset composition would have application in wire and cable." Applicant respectfully disagrees that he took this position. Applicant stated that Kim et al. if attempted to be employed as a thermoplastic (modified) would not be suitable. He followed this with a discussion on why a modified thermoplastic Kim et al. used as a insulation would not find application.

Re claim 43.

Applicant has added definition to this claim showing that applicant's propylene copolymer is substantially distinct from the ethylene- propylene elastomer mentioned in Kim et al.

Comments on Allowable Subject Matter

Claims 40 and 41 now deemed to be dependent on an allowable base claim were not amended.

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Conditional Request for Constructive Assistance

Applicant has amended the claims of this application so that they are proper, definite, and define novel and unobvious matter. If, for any reason this application is not believed to be in full condition for allowance, applicant respectfully requests the constructive assistance and suggestions of the Examiner pursuant to M.P.E.P. 707.07(j) in order that the undersigned can place this application in allowable condition as soon as possible and without the need for further proceedings.

Very Respectfully,


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These search terms have been highlighted: **polypropylene radiation crosslinking**

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Radiation Processing for Modification of Polymers

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Abstract

Radiation processing has been demonstrated on a large commercial scale to be a very effective means of improving end-use properties of various polymers. It is a well-established and economical method of precisely modifying the properties of bulk polymer resins and formed polymer components. The reactions of cross-linking, degradation and grafting on polymers initiated by **radiation** have found many useful applications in plastic and rubber materials. Important properties of polymer materials, such as mechanical properties, thermal stability, chemical resistance, melt flow, processability and surface properties can be significantly improved by **radiation** processing. Modern high energy, high-power electron accelerators have made industrial irradiation processes attractive with high throughput, low cost and other advantages over conventional chemical processes such as lower emissions of volatile organic compounds (VOCs), better energy utilization and precise process control. In this paper, the basic principles of and main applications for **radiation** processing of polymers are reviewed. The prospects for new applications of this technology in the future are also discussed.

Introduction

Radiation processing using gamma rays, electron beams (EB) or X-rays has been demonstrated on a large commercial scale to be a very effective means of improving end-use properties of various polymers. Practical applications for **radiation** processing of materials have been evolving since the introduction of this technology nearly fifty years ago. The main applications are for modification of polymer materials through **radiation crosslinking**, degradation and grafting. For irradiation technologies, the main irradiating sources include gamma rays from radioactive isotopes such as cobalt 60, electron beams from electron accelerators, and X-rays converted from electron beam. **Radiation** processing of polymer materials involves subjecting the polymers to the irradiation, usually in a continuous mode for industrial modifications. In this review, the basic principles of and main applications for **radiation** processing of polymers are discussed. The prospects for new applications of this technology in the future are also reviewed.

Radiation Chemistry of Polymers

When the **radiation** from a gamma ray, EB or X-ray source interacts with a polymer material, its energy is absorbed by the polymer material and active species such as radicals are produced, thereby initiating various chemical reactions. There are three fundamental processes that are the results of these reactions: (1) **crosslinking**, where polymer chains are joined and a network is formed; (2) **degradation**, where the molecular weight of the polymer is reduced through chain scissioning; and (3) **grafting**, where a new monomer is polymerized and grafted onto the base polymer chain. When monomers are irradiated, polymerization can also be initiated. **Radiation** "curing" (as in the case of coatings or composites), for example, is a combination of polymerization and cross-linking. Different polymers have different responses to **radiation**. A parameter called "G value" is usually used to quantify the chemical yield resulting from the **radiation**. G value is defined as the chemical yield of

radiation in number of molecules reacted per 100 eV of absorbed energy. G-values for **crosslinking** G(X) and for chain scission G(S) for some of the common polymeric materials can be found in many references ^(1,2) Materials with G(S)/G(X) ratios smaller than 1.00 are favored for cross-linking. Materials with G(S)/G(X) ratios greater than 1.00 tend to undergo degradation more. Materials whose G(X) and G(S) values are both low are more resistant towards **radiation**. The different responses to **radiation** for different polymers are intrinsically related to the chemical structures of the polymers. ~~Polymers with more hydrogen atoms on the side (e.g., polyethylene) tend to cross-link with radiation. Polymers with a methyl group (e.g., polypropylene), di-substitutions (e.g., polymethacrylate) and per-halogen substitutions (e.g., polytetrafluoroethylene) would more likely undergo degradation with radiation.~~ Aromatic polymers with benzene rings either in the main chain or on the side (e.g., polystyrene and polycarbonate) are usually **radiation** resistant. Cross-linking and degradation (through chain scission) are two competing processes that always co-exist under **radiation**. The overall effect depends on which of the two is predominant at a certain time. It should also be kept in mind that for a given polymer G(X) and G(S)

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both change with **radiation** conditions such as the absorbed dose and the temperature. Both G(X) and G(S) would increase with the increase of the dose. However, G(S) for a polymer generally increases more than G(X) does with increasing dose. Therefore, when G(X) of a polymer is much greater than G(S), the MW would continuously increase due to continuous cross-linking, but the MW will level off because G(S) will increase faster. When G(X) is greater, but not much greater than G(S), G(S) will eventually catch up with G(X), and the MW will show a turning point, with the overall reaction changing from cross-linking to degradation. Continuous degradation will occur when G(S) is greater than G(X). G(X) and G(S) are also dependent on irradiation conditions, such as temperature and atmosphere. For some polymers, elevated temperature may increase the mobility of the polymer chains and make it more favorable to cross-linking. Oxygen in the air usually assists the degradation more through a peroxide radical mechanism, so an oxygen-free atmosphere would usually be more favorable for cross-linking.

Radiation Processing and Its Industrial Applications

Cross-linking

Crosslinking is the most important effect of polymer irradiation and has the larger number of applications because it can usually ~~improve the mechanical and thermal properties and chemical, environmental and radiation stabilities for both preformed parts and bulk materials.~~ The following are some examples of applications. (1) ~~Wire and Cable. The crosslinking of insulation on electrical wires and cables was one of the first practical applications of radiation processing.~~ Polymers used in this application include polyethylene, polyvinylchloride, ethylene-propylene rubber, polyvinylidene fluoride, and ethylene-tetrafluoroethylene copolymers. ~~Product improvements~~ obtained by irradiation include increased tolerance to high temperature environments and overloaded conductors, fire retardation, increased abrasion resistance and tensile strength, reduction in cold flow, increased resistance to solvents and corrosive chemicals as well as some other important characteristics. Irradiated wires are commonly used in automobiles, military vehicles, aircraft, spacecraft and many other applications where high performance is required. (2) Heat-Shrinkable Products. Thin-walled plastic tubing and plastic films are cross-linked to obtain the so-called "memory" effect from the cross-linked network. **Radiation** cross-linking fixes or stabilizes the original dimensions of the tubing or films. When the material is heated above the temperature where the unirradiated material would melt, it becomes elastic and can be expanded to at least twice its original dimension. When cooled, it maintains the expanded dimension but retains the "memory" of its original dimension. When heated again, it contracts to the original dimension. Polyethylene is commonly used for this application ⁽⁴⁾. Many commercial products have been developed. Some examples for tubing products are encapsulations for electronic components, bundles of electrical wires and exterior telephone cable connectors. The applications for films are mainly in the food industry, where heat-shrinkable wrapping material is used to make attractive, sealed packages. Modern packaging films use blends of several different polymers to provide

[3]

desirable properties like clarity, toughness, oxygen exclusion and moisture retention. (3), Rubber Tires. Automobile tire tread sections are irradiated to obtain partial cross-linking before the tire is assembled. This stabilizes their thickness during the final thermal curing process. It also prevents the steel belt from migrating through its supporting rubber layer. The result is a higher quality tire with more uniform thickness and better balance. This allows the tire to be made thinner to save material and reduce cost. A thinner tire also generates less frictional heating on the road. Dose requirements are in the range of 30 to 50 kGy^[2, 5]. (4) Plastic Pipes. Cross-linked plastic pipes are used to distribute potable hot water and for floor and wall heating applications. The pipe may either be made entirely of polyethylene, or may be a composite pipe consisting of an inner layer of polyethylene to keep the water from contacting a middle layer of thin aluminum, which resists the water pressure, and an outer layer of polyethylene for abrasion resistance. Both the inner and outer plastic layers are irradiated simultaneously with electron beam. Cross-linking enhances the mechanical properties and the thermal stability of the pipes. Plasticized PVC pipes are also cross-linked with radiation^[6]. (5) PTC ("Positive Temperature Coefficient") Materials. When polymers such as polyethylene and ethylene-vinyl acetate copolymers (EVA) are filled with conductive fillers such as carbon black, conductive composite materials are formed. These materials can be cross-linked by radiation to obtain a useful property called the "PTC effect". The PTC effect refers to the sudden drastic increase of electric resistivity of the material upon reaching a certain temperature when the material is heated. The resistivity can increase by many orders of magnitude. This property can be employed to make self-limiting heating cables, resettable microfuses, etc. Upon reaching the critical temperature, the electric circuit would be broken, and the material would stop heating itself^[7-8]. (6) Plastic Foams. Plastic foam is made

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by mixing a foaming compound with the basic polymer and then heating the mixture to release gas bubbles emanating from a nitrogen-containing foaming agent in the plastic material. Polyethylene, ethylene vinylacetate copolymer and polypropylene are suitable materials. A typical foaming agent is azodiocarbonamide or nitrogen. Radiation crosslinking allows the use of higher expansion temperatures and simplifies the control of the expansion process. Applications include foamed insulation in coaxial cables, gaskets, coated tapes, floor backing, helmet liners, athletic safety pads, bra cups, automobile seat padding and jewelry case liners^[9]. (7) Orthopedic Devices. Orthopedic devices such as hip joints are usually made of ultra high molecular weight polyethylene (UHMWPE). Radiation cross-linking can significantly improve the wear resistance of the surface these devices. When irradiated in air, oxidation and oxidative degradation during the irradiation can cause surface and sub-surface wear leading to delamination or deep pitting and deterioration of mechanical properties. This problem can be solved by carrying out the irradiation with the polymer in melt state, using an oxygen-free atmosphere, using shields or masks to expose only part of the surface to irradiation and thermal stabilization or annealing, etc.^[10-11] (8) Hydrogels. A hydrogel is a polymeric material which has the ability to swell in water and retain a significant fraction (more the 20%) of water within its structure without being dissolved in water. The hydrogel network can be formed by radiation crosslinking. Some polymers that can form hydrogels are polyvinylalcohol (PVA), polyacrylamide (PAAm), polyvinyl-pyrrolidone (PVP), polyethylene oxide (PEO) and methyl cellulose (MC). Hydrogels have excellent biocompatibility and can be used for wound dressing, controlled release of drugs, and enzyme supports^[12-13].

Curing

Curing is a combination of polymerization and cross-linking initiated by radiation from monomers and oligomers. Major advantages of radiation curing include reduction or elimination of volatile organic compounds (VOCs) and faster curing. Radiation curing of coatings, inks and thin adhesives usually only requires low energy electron beams. The following are applications for medium to high beam energies. (1) Composite Materials. Advanced composites, such as carbon fiber reinforced polymer resins, have become very important materials that are used for a wide range of applications because of their excellent mechanical properties and low weight. Conventionally, the composites have been cured by thermal means, which employ autoclaves or ovens and high temperature to

accomplish the chemical reactions of curing (i.e., polymerization and cross-linking of the polymer resin). Research on EB curing of composites dates back to late 1960's, but it was in the 1990's that a large volume of research and development was conducted, primarily through the co-operation of national labs, the aerospace industry, and academic institutions (e.g., CRADAs, Cooperative Research And Development Agreements). EB curing has drawn such high interest because of its many important advantages over the traditional thermal curing, e.g., faster cure time, lower (ambient) temperature curing (which significantly reduces internal stress), capability to cure large parts, simplified tooling, improved material handling, reduced VOC emission and better process control. Typical resins are acrylated epoxies and EB curing may be accomplished with either a free-radical mechanism (no initiator needed) or a cationic mechanism (initiator required). Doses are typically in the range of 100 to 200 kGy. The main potential applications are in automotive and aerospace industries ^[14-16]. Although industrial applications have yet to be widely commercialized due to some property drawbacks, such as poor resin-fiber interfacial adhesion and poor interlaminar shear strength, progress is being made to overcome these remaining deficiencies. (2) Adhesive Bonding of Thick Components. There have already been examples of EB curing of adhesives for the bonding of large composite structures for aerospace and automotive applications ^[17-18]. Compared with conventional thermal curing, EB curing of adhesives has many environmental and processing advantages such as reduction or even elimination of organic solvents (VOCs), faster curing, elimination of time constraints (due to the long life of EB-curable adhesives), fewer processing steps, reduced cost, elimination of autoclave/oven processing, reduced residual stress, prevention of de-bonding of dissimilar materials and "spot-welding" type of bonding by beaming at specific areas.

Degradation

Some polymers that undergo degradation upon EB processing include polytetrafluoroethylene (PTFE), polypropylene (PP) and cellulose. Although degradation usually brings about deterioration of mechanical properties of polymers and needs to be avoided in many cases (e.g., radiation sterilization), some good applications have been found for chain scission of polymers by radiation. (1) Particle Size Reduction for Fine Powders. A well-known example of applications for degradation is the making of fine PTFE powders. PTFE has a high G(S) and can be readily degraded to lower molecular weight by radiation. One of the results of the

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degradation of PTFE is the much higher brittleness of the material. This effect is used to convert scrap PTFE into fine particles or micronized powder. The unirradiated scrap is too tough, doughy and slippery to grind, but the irradiated material can be ground readily. The dose requirements can be very high, in the range of 500 to 1000 kGy, although doses as low as 50 kGy are also used for some specific applications ^[19-21]. (2) Melt Flow Rate Adjustment. Another effect of degradation is the increase of the melt flow rate (MFR) of the polymer. In particular, polymers can be intentionally degraded by radiation in air to improve the processability for extrusion, etc. Irradiated polymers can be blended with unirradiated polymers to adjust the melt flow and improve the processability, for example producing a range of MFR for PTFE that it is high enough for the polymer to be melt-processable but low enough so that the polymer is too brittle to process ^[22]. Irradiated PP can be mixed with unirradiated PP, and the degree of degradation may be controlled so that mechanical properties are not significantly deteriorated, yet melt flow advantages are obtained ^[23]. (3) Compatibility Improvement with Oxidation. When polymers such as PTFE are irradiated in air, the oxygen and moisture in the air cause oxidation in addition to degradation. The polar functional groups such as the carboxylic acid groups on PTFE can help improve the compatibility of PTFE with other polymers.

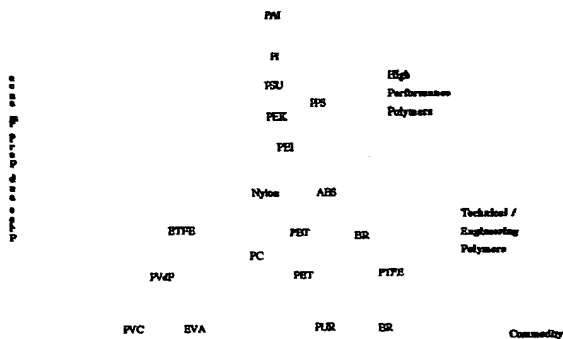
Grafting

Radiation-initiated grafting is known to be a very good method for surface modification of polymer materials. The surface properties of polymers can be modified by graft copolymerization with different monomers. Grafting can be accomplished by irradiation on common polymers such as polyethylene, polypropylene and

fluoropolymers. Most work has been done on polymer films, membranes, fibers and natural and synthetic textiles. Examples include grafting of acrylonitrile, maleic anhydride, styrene, N-vinylpyrrolidone acrylic acid and various acrylate monomers on polyethylene and fluoropolymers. Other examples are the bonding of styrene on cellulose, vinylpyridines on wool and p-nitrostyrene on polyethylene, **polypropylene** and polyvinyl chloride Hydrophilic properties can be imparted to hydrophobic polymers [24]. Biocompatibility of various polymers can be improved in this way for medical uses [26]. Ion exchange membranes and fuel cell/battery separator films can be made by grafting styrene onto porous fluoropolymer (e.g., polyvinylidene fluoride) membranes followed by sulfonation [27]. Other applications include permeation separation membranes, surface adhesion promotion, chelating fibers for sea water treatment and recovering of precious metals, etc. Dose requirements may be less than 10 kGy. Much of the research in this area has been done outside the U. S., and there has been no large-scale commercialization yet.

New Areas of Applications in the Future

Although a great deal of research and development for **radiation** processing of polymers have been done in the past fifty years, and there have been many successful commercialized applications, this versatile technology still has potential for new areas of applications in the future. Some of the good applications from past development still need to overcome barriers for real commercialization. New technologies are raising new requirements for new polymer materials with even better properties and lower cost for producing them. With the emergence of new irradiator technologies, some of the applications that were not feasible in the past may become practical now. Increased environmental consciousness may also open up some doors for new applications. As a very useful means of modifying polymer materials, **radiation** processing can be a good economical way to “upgrade” the low-price commodity polymers to high value specialty polymers, engineering polymers and high performance polymers (Figure 1). The following are some of the potential new areas of industrial applications for the near future.



PE

NR

BPM

ES

IP

Polymers

Radiation Cross-linking

Radiation Degradation

Figure 1. Classification of Polymers

Cross-linking

Improvements in mechanical properties and thermal stability of formed parts is very feasible through cross-linking. **Radiation** cross-linking is a very clean, easy and cost-effective process for formed parts. On bulk polymers, **crosslinking** of engineering polymers (such as polysulfones^[28]) can also be employed for further performance enhancement. **Radiation** cross-linking of traditionally degradation type polymers, such as PTFE and polypropylene (especially certain types of PP^[30]), may also find useful applications in the future.

[29]

Grafting

A significant amount of research and development work has been done in the past on **radiation** grafting for surface modification. However, to date few of the applications have been commercialized. This is at least partly due to the significantly more complex process of **radiation** grafting, which usually requires handling of solvent(s) and non-ambient atmosphere. Successful commercialization will require engineering effort to overcome this obstacle. Although most of the work for **radiation** grafting has been done onto polymer membranes, films and fibers, there have also been research results and patents on grafting onto bulk polymers such as powders. Modification of bulk polymer properties may open a new area for **radiation** grafting applications. Grafting may also be done on inorganic fillers and other inorganic materials for compatibility enhancement and other property modification.

Blending

Radiation processing may also play a role in the blending of different polymers and polymer resins and inorganic fillers. Compatibility of the different materials is usually an issue for blending. Grafting and intentional surface oxidation can be employed to enhance the compatibility. Examples include grafting on fluoropolymer powders for blending with polyamides and **radiation** oxidation of polyethylene for blending with inorganic fillers

[31]

Environmental Applications

In this environment-conscious age, more and more stringent environmental regulations have great impact on uses of polymer materials. Besides the advantage of reduction or elimination of VOCs for **radiation** curing of coatings, composites and adhesives, there are other areas that **radiation** processing can help with environmental applications. Because of the increasing concerns about the health hazards of acrylates, "non-acrylate" curing has been explored for using charge transfer (CT) complexes as monomers to replace traditional acrylates employing a cationic curing mechanism^[32]. **Radiation** cross-linking and grafting may be able to help improve VOC permeation resistance for fuel containers, etc. **Radiation** degradation can be employed for reclamation of recycled plastics and rubbers^[33]. Compatibility enhancement by **radiation** processing can also play an significant role in polymer recycling^[34].

Biomaterials

Besides the **radiation** cross-linking of hydrogels and orthopedic devices, **radiation** processing can have other applications in the biomaterials area. For example, biocompatible materials can be greatly enhanced by **radiation** grafting of special functional groups, usually followed by further bonding to biological molecules^[35-36]. A very